DN 3674
Patents

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of

Yoshikazu Matsumoto

Serial No.: 10/039,664

Filed: January 4, 2002

For: FORMING MATERIAL

Examiner: Elizabeth M. Cole

Art Unit: 1771

Last Office Action Mailed April 20, 2004

Commissioner for Patents
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Alexandria, Virginia 22313-1450

CERTIFICATE OF MAILING

I hereby certify that this Declaration Under Rule 132, along with any paper or document referred to herein as being attached or enclosed, is being deposited in the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450 on

Aug. 19, 2004.

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DECLARATION UNDER RULE 132

Dear Sir:

I, Yoshikazu Matsumoto declare as follows:

1. I am a co-inventor of the subject matter of the above-identified patent application. I received the following degrees in the following years from the following institutions:

Degree	Year	Institution
Bachelor of science	March 1997	Faculty of Engineering, Chiba Institute of Technology
Master of science	March 1999	Faculty of Engineering, Chiba Institute of Technology

2. Since April 1999, I have been employed as follows in research and development of fixing materials for orthopaedic surgery, particularly research and develop-ment of urethane resins and composite materials:

ALCARE Co., Ltd., 21-10, 1-chome, Kyojima, Sumida-ku, Tokyo, Japan

3. The test samples run and reported at table I on page 18 of the specification of the present application were conducted under my direction and control. The curing times were as shown ranging between about 4 minutes 40 seconds and 6 minutes and 10 seconds, or approximately from about 5 minutes to about 6 minutes. In those examples, the *tack free* time was from 4 min 20 sec (260 sec) to 5 min 30 sec (330sec).(Refer to TABLE (1) attached.) The tack free time was measured in accordance with JIS–A 5758 (1986), by which Aoki measured his tack free time in his patent.

4. I have read and I am very familiar with the subject matter disclosed and claimed in U.S. Patent 4,880,869 by Masaaki Aoki, et al. Although their disclosure relates to certain moisture curable polyurethane compositions and their applications, the compositions are restricted to those developed from β-amino-β-propiolactam derivatives. Such compositions are made from a different reaction mechanism than my invention. My reaction mechanism is characterized by addition of water (moisture in the atmosphere) to an isocyanate radical of the urethane prepolymer, subsequent decarboxylation of the resultant adduct to form an amine, and a reaction of the amine with other isocyanate radicals of the urethane prepolymer to carry out curing. (Refer to Scheme (1) attached.) Whereas in the Aoki, the β-amino-β-propiolactam derivative first reacts with water to form two secondary amine radicals, which then react with an isocyanate radical of the urethane prepolymer.

5. In the forming material of the present invention, the NCO radical content is about 1-5% by weight. This is one feature of the present invention. The reason is as follows.

The NCO radical content is the total of the NCO radicals contained in the urethane prepolymer and those, left unreacted with the polyol component, contained in the polyisocyanate component used for preparing the prepolymer.

In the preparation of the urethane prepolymer, two moles of the polyisocyanate component react with one mole of the polyol component. Therefore, when the molar ratio of the polyisocyanate component to the polyol component is more than 2, unreacted (free) polyisocyanate exists in the forming material. A free polyisocyanate reacts not only with the urethane prepolymer but also with another free polyisocyanate to raise the ratio of compounds having the rigid structure such as a urea bond and a diphenylmethane skeleton, which results in poor molding property. (Refer to Scheme (2) attached.) As the amount of the free isocyanate radicals increases, the ratio of the rigid structure gets higher thus deteriorating the molding property.

In the example 1, the polyisocyanate component to polyol component molar ratio is 1.96. In the comparative example 1, the molar ratio is 3.04. Thus, the molding property in the example 1 is good whereas the molding property in the comparative example 1 is not so good.

6. The compositions of my invention are distinctly different from those compositions emanating from amines, because my compositions, unlike Aoki et. al, are characterized with the critical properties of excellent molding properties with short curing times and which properties can readily be identified in our specification at page 18 Table I. When prepolymers consist essentially of polyisocyanate and a polyol constituent, without the β-amino derivatives, as do our compositions, and contain terminal isocyanate radicals having a total NCO weight percent from 1 to 5 % and cure from about 5 minutes to about 6 minutes, the tack free time is only 4 min 20 sec (260 sec)

to 5 min 30 sec (330sec). The tack free time however in U.S. Patent 4,880,869 at example 14 Table 3 even where the terminal isocyanate radical content is 1.9 % by weight is at least 23 minutes.

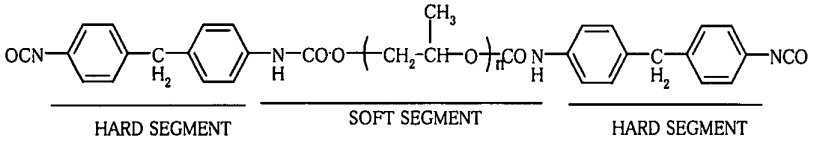
7. I further declare that all statements made here and of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of title 18 of the United States code; and that such willful false statements may jeopardize the validity of the above-referenced application or any patent issuing thereon.

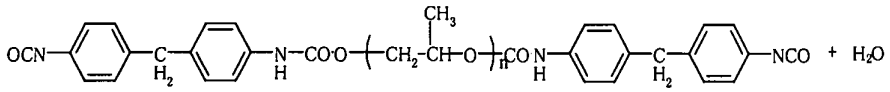
August 16, 2004
Date

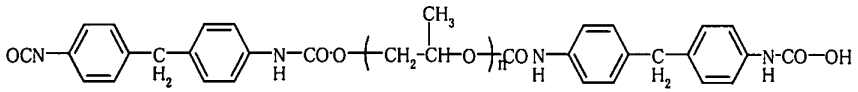
Yoshikazu Matsumoto
Signature

SCHEME(1) : REACTION MECHANISM OF THE PRESENT INVENTION

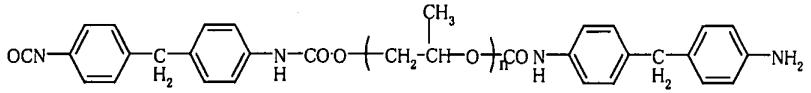
PREPOLYMER





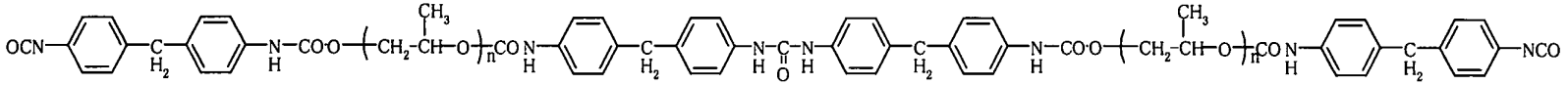


DECARBOXYLATION

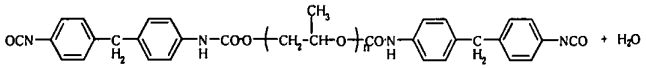


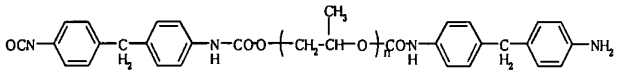
(1)

+PREPOLYMER



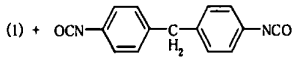
SCHEME(2) : REACTION OF FREE POLYISOCYANATE (DIPHENYL METHANE DIISOCYANATE)

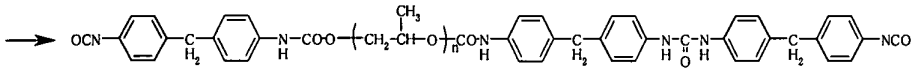


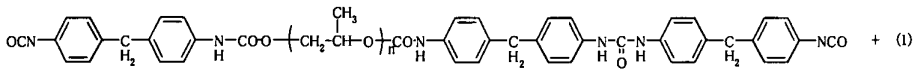


(1)

*REACTION OF FREE POLYISOCYANATE WITH URETHANE PREPOLYMER

(1) +  [MDI]





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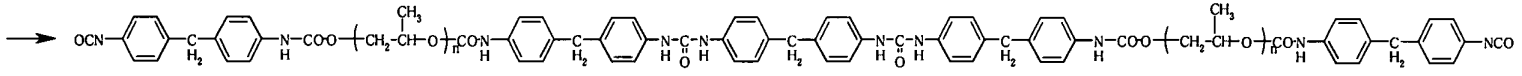
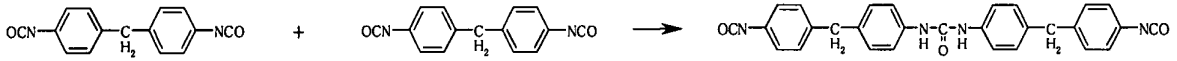


RIGID STRUCTURE

*REACTION OF FREE POLYISOCYANATE WITH ANOTHER FREE POLYISOCYANATE



DIPHENYL METHANE SKELETON

Table (1)

	Example1	Example2	Example3	Example4	Example5	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4
Polyol Component									
PPG−2000	750. 0	750. 0	750. 0			657. 0	657. 0	700. 0	657. 0
PPG−1000	−	−	−	740. 0	−	−	−	−	−
PPG−4000	−	−	−	−	815. 0	−	−	−	−
Polyisocyanate Component	184. 0	215. 0	230. 0	260. 0	185. 0	250. 0	321. 0	219. 0	321. 0
Catalyst	60. 0	65. 0	70. 0	60. 0	60. 0	60. 0	70. 0	60. 0	20. 0
NCO Content(%)	3. 3	4. 0	4. 5	2. 4	4. 2	6. 0	8. 0	9. 4	8. 0
Catalyst Content(%)	5. 9	6. 3	6. 6	5. 6	5. 6	6. 2	6. 7	6. 1	1. 9
Viscosity(Pa・s)	30. 4	29. 2	28. 6	50. 0	8. 0	26. 6	24. 0	21. 5	24. 0
Curing Time(sec)	330	310	280	300	370	310	270	270	≧600
Tack Free Time(sec)	300	280	260	330	300	−	−	−	−
Molding Property	○	○	○	○	○	△	×	×	×